BRITISH STANDARD

BS 553:1990

Specification for

Ethyl acetate for industrial use

Confirmed
January 2011

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/51, upon which the following bodies were represented:

British Pharmacopoeia Commission
British Society of Perfumers
Chemical Industries Association
Solvents Industry Association Ltd.

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Contents

Foreword

This British Standard, which has been prepared under the direction of the Chemicals Standards Policy Committee, supersedes BS 553:1965 which is withdrawn. In this revision of BS 553 the requirements for colour, residue on evaporation and acidity have each been made more stringent and the requirement for relative density has been replaced by one for density at 20 °C. In addition, some of the appendices describing test methods in the previous edition have been replaced by references to general test methods.

This British Standard is related to ISO 1386:1983 but is not equivalent in technical content. ISO 1386 is published by the International Organization for Standardization (ISO). ISO 1386 lists various general test methods, which correspond to those to which reference is made in this British Standard, and it also describes methods for the determination of acidity and for the determination of ester content which are similar to those described in Appendix B and Appendix C, respectively, of this British Standard. ISO 1386 does not specify limits for any of the properties of ethyl acetate or other solvent acetates.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This British Standard specifies requirements for ethyl acetate suitable for industrial purposes.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Description

The material shall be clear and free from matter in suspension, as assessed by visual inspection, and shall consist essentially of the ethyl ester of acetic acid ($CH_3COOC_2H_5$).

3 Sampling and size of sample[1)]

A representative sample of the material measuring not less than 0.5 L shall be taken from the bulk for the purpose of examination in accordance with this standard. The sample shall be placed in a clean, dry and air-tight, ground glass-stopped bottle, or screw-capped bottle fitted with a polyethylene cone insert, of such capacity that it is almost filled by the sample.

NOTE 1 When it is necessary to seal the container, care should be taken to avoid the risk of contaminating the contents in any way.

NOTE 2 Sufficient ullage should be left in the bottle to avoid excessive pressure changes that could arise from temperature variations during storage and handling. About 10 % ullage is recommended.

4 Colour

The colour of the material shall not exceed 10 Hazen colour units when measured by the method described in BS 5339.

5 Density

The density of the material at 20 °C, measured by the method described in BS 4522, shall be not lower than 0.898 g/mL and not higher than 0.901 g/mL.

6 Distillation range

When the material is distilled by the method described in BS 4591, modified as described in Appendix A, the initial boiling point at 101.3 kPa pressure shall be not lower than 76.5 °C and the dry point at 101.3 kPa pressure shall be not higher than 78.5 °C.

7 Residue on evaporation

The residue on evaporation of the material shall not exceed 0.0020 % (*m*/*m*) when determined by the method described in BS 4524.

8 Water content

The material shall not contain more than 0.10 % (*m*/*m*) of water when determined by the method described in clause **2** of BS 2511:1970 using 20 mL of the material.

9 Acidity

The acidity of the material, calculated as acetic acid (CH_3COOH), shall not exceed 0.0050 % (*m*/*m*) when determined by the method described in Appendix B.

10 Ester content

The material shall contain not less than 99.0 % (*m*/*m*) of esters, calculated as ethyl acetate ($CH_3COOC_2H_5$), when determined by the method described in Appendix C.

[1)] Detailed information on the sampling of liquid chemical products is given in BS 5309-1 and BS 5309-3.

Appendix A Determination of distillation range

Determine the distillation range, in terms of the initial boiling point and the dry point, by the method described in BS 4591, using the following thermometer and temperature corrections.

a) *Thermometer* (**5.1.2** of BS 4591:1990). Use a thermometer designated F100C/100 complying with BS 593.

b) *Distillation* (**7.2** of BS 4591:1990). Regulate the rate of heating so that the first drop of distillate falls from the end of the condenser after 7 min to 12 min.

c) *Corrections to be applied to observed temperatures* (**9.1.2** of BS 4591:1990). If the corrected barometric pressure deviates from 101.3 kPa apply corrections to the observed temperatures by subtracting 0.031 °C for every 0.1 kPa above 101.3 kPa, or adding 0.031 °C for every 0.1 kPa below 101.3 kPa.

NOTE These corrections are valid only for pressures above 93.3 kPa.

Appendix B Determination of acidity

B.1 Principle

A test portion is diluted with 95 % (*V*/*V*) ethanol (or methylated spirits) and titrated with standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

B.2 Reagents

B.2.1 *General*

During the analysis, use only reagents of recognized analytical grade, only methylated spirits complying with BS 3591, and only water complying with grade 3 of BS 3978.

B.2.2 *Ethanol or industrial methylated spirits, 95 % (V/V)*

NOTE The use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative.

B.2.3 *Sodium hydroxide,* standard volumetric solution, c(NaOH) = 0.100 mol/L.

B.2.4 *Phenolphthalein*, 5 g/L ethanolic solution. Dissolve 0.5 g of phenolphthalein in 100 mL of the ethanol or industrial methylated spirits (**B.2.2**) and add the sodium hydroxide solution (**B.2.3**) until a pale pink coloration is obtained.

B.3 Apparatus

B.3.1 *Ordinary laboratory apparatus*

B.3.2 *Conical flask*, of 250 mL capacity, of borosilicate glass, fitted with a ground glass stopper carrying a guard tube containing sodium hydroxide on an inert support (soda lime).

B.3.3 *Burette*, of 10 mL capacity, graduated in 0.02 mL divisions, complying with class A of BS 846.

B.4 Procedure

B.4.1 ***Test portion***

Take 50 ± 0.5 mL of the sample, measured at 20 °C.

B.4.2 ***Determination***

Place 50 mL of the ethanol or industrial methylated spirits (**B.2.2**) in the conical flask (**B.3.2**), add 0.5 mL of the phenolphthalein solution (**B.2.4**) and add the sodium hydroxide solution (**B.2.3**) until a pale pink coloration is obtained. Add the test portion (**B.4.1**), mix and titrate the mixture with the sodium hydroxide solution, using the burette (**B.3.3**), until a pink coloration, persisting for at least 10 s, is obtained. Stopper the flask and swirl its contents after each addition of sodium hydroxide solution.

B.5 Expression of results

The acidity A, expressed as a percentage by mass of acetic acid (CH_3COOH), is given by the equation:

$$A = \frac{0.006\ V_1}{\rho \times 50} \times 100$$

i.e.

$$A = \frac{0.012\ V_1}{\rho}$$

where

V_1 is the volume of the sodium hydroxide solution used for the determination (in mL);

ρ is the density of the sample at 20 °C (determined by the method described in BS 4522) (in g/mL);

0.006 is the mass of acetic acid corresponding to 1.00 mL of sodium hydroxide solution, c(NaOH) = 0.100 mol/L (in g).

Report the result to two significant figures.

B.6 Test report

The test report shall include the following information:

a) a complete identification of the sample;

b) a reference to the method used, i.e. BS 553 Appendix B;

c) the results expressed in accordance with **B.5**;

d) any unusual features noted during the determination;

e) any operation not included in this appendix or regarded as optional.

Appendix C Determination of ester content

C.1 Principle

The esters present in a test portion are saponified with excess ethanolic potassium hydroxide solution and the excess is determined by titration with standard volumetric hydrochloric acid solution, using phenolphthalein as indicator.

C.2 Reagents

C.2.1 *General*

During the analysis, use only reagents of recognized analytical grade, only methylated spirits complying with BS 3591, and only water complying with grade 3 of BS 3978.

C.2.2 *Potassium hydroxide,* 56 g/L solution in 95 % (*V*/*V*) ethanol or 95 % (*V*/*V*) industrial methylated spirits.

NOTE The use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979. Section 10, for purposes for which industrial methylated spirits is an acceptable alternative.

C.2.3 *Hydrochloric acid*, standard volumetric solution, *c*(HCl) = mol/L.

C.2.4 *Phenolphthalein,* 5 g/L ethanolic solution. Prepare as described in **B.2.4**.

C.3 Apparatus

C.3.1 *Ordinary laboratory apparatus.*

C.3.2 *Two conical flasks,* of borosilicate glass, capacity 250 mL, fitted with ground glass stoppers.

C.3.3 *Two reflux condensers,* water-cooled, with ground glass joints to fit the conical flasks (**C.3.2**).

C.3.4 *Weighing pipette,* capacity 10 mL.

C.4 Procedure

C.4.1 ***Test portion***

Using the weighing pipette (**C.3.4**), transfer between 2.0 g and 2.4 g of the sample, weighed to the nearest 0.001 g, to a conical flask (**C.3.2**) containing 50.0 mL of the potassium hydroxide solution (**C.2.2**), introduced by means of a one-mark pipette.

C.4.2 ***Blank test***

Introduce 50.0 mL of the potasium hydroxide solution (**C.2.2**), using the same one-mark pipette referred to in **C.4.1**, into the second conical flask (**C.3.2**).

C.4.3 ***Determination***

Attach a reflux condenser to each conical flask and heat the two flasks for 1 h in a boiling-water bath. Withdraw the flasks, still carrying their condensers, and immerse them in cold running water. When cool, wash down the inside of each condenser with two 20 mL portions of water, collecting the washings in the flask. Disconnect the flasks and wash each joint with a further 20 mL of water, again collecting the washings in the appropriate flask.

Add 0.5 mL of the phenolphthalein solution (**C.2.4**) to each flask and immediately titrate the contents of each flask in turn with the hydrochloric acid solution (**C.2.3**) until the pink colour is just discharged.

C.5 Expression of results

The ester content E, expressed as a percentage by mass of ethyl acetate ($CH_3COOC_2H_5$), is given by the equation:

$$E = \frac{88.1\ (V_0 - V_1)}{1000 \times m} \times 100$$

i.e.

$$E = \frac{8.81\ (V_0 - V_1)}{m}$$

where

V_0 is the volume of the hydrochloric acid solution used for the titration of the blank (in mL);

V_1 is the volume of the hydrochloric acid solution used for the titration of the test solution (in mL);

m is the mass of the test portion (in g);

88.1 is the relative molecular mass of ethyl acetate.

Report the result to one decimal place.

C.6 Test report

The test report shall include the following information:

a) a complete identification of the sample;

b) a reference to the method used, i.e. BS 553 Appendix C;

c) the results expressed in accordance with **C.5**;

d) any unusual features noted during the determination;

e) any operation not included in this appendix or

Publications referred to

BS 593, *Specification for laboratory thermometers.*

BS 846, *Specification for burettes.*

BS 2511, *Methods for the determination of water (Karl Fischer method).*

BS 3591, *Specification for industrial methylated spirits.*

BS 3978, *Specification for water for laboratory use.*

BS 4522, *Method for determination of absolute density at 20 °C of liquid chemical products for industrial use.*

BS 4524, *Method for determination of residue on evaporation on a water bath.*

BS 4591, *Method for determination of distillation characteristics of organic liquids (other than petroleum products).*

BS 5309, *Methods for sampling chemical products.*

BS 5309-1, *Introduction and general principles.*

BS 5309-3, *Sampling of liquids.*

BS 5339, *Method of measurement of colour in Hazen units (platinum-cobalt scale) of liquid chemical products.*

ISO 1386, *Solvent acetates for industrial use — Methods of test*[2)].

[2)] Referred to in the foreword only.

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